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Title: Moisture Studies of PBX 9501

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NATIONAL LABORATORY

Moisture studies of PBX 9501

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LANL Energetic Materials Review
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Abstract

Pellets of PBX 9501 were first dried for 3 months to remove residual moisture, then stored in different humidity levels for 41 days while monitoring the moisture uptake by measuring the change in mass of the pellets. Several reference pellets were left in the dry environment, and one sample that had been conditioned to a high moisture content was changed back to a dry environment. A standard compression test was done on these pellets to determine the effects of the moisture content on the mechanical properties. The load deflection behavior did not change character, but the peak stress decreased noticeably with increasing moisture content.

Results from earlier tests on other variables are reviewed, and recommendations for improving surveillance testing techniques are presented. Preliminary results from a continuing study on reduced NP properties are shown. Future goals for mechanical testing are outlined and discussed, and connections are made with the LDRD project on Polymer Aging.

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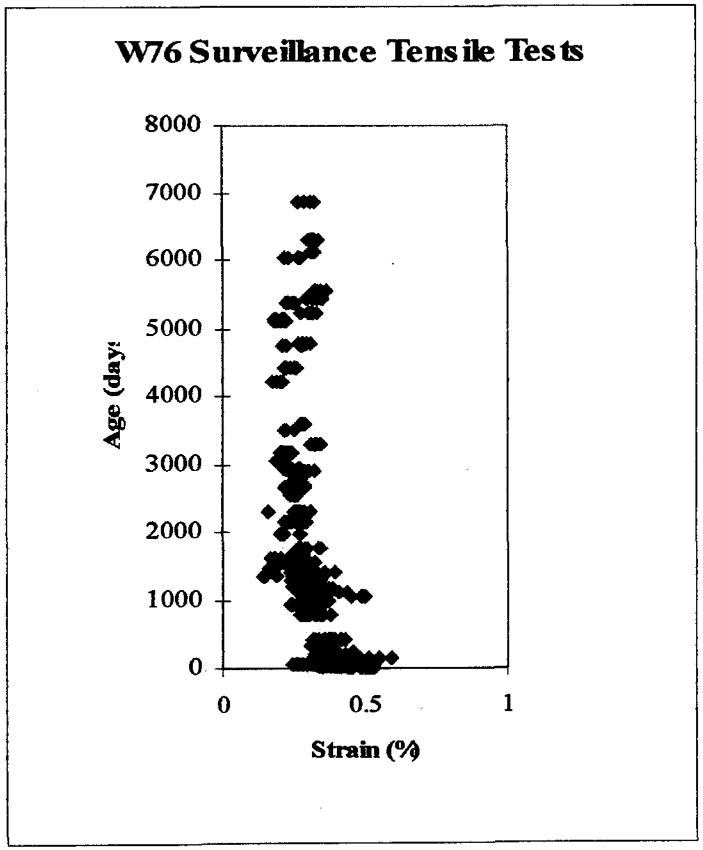
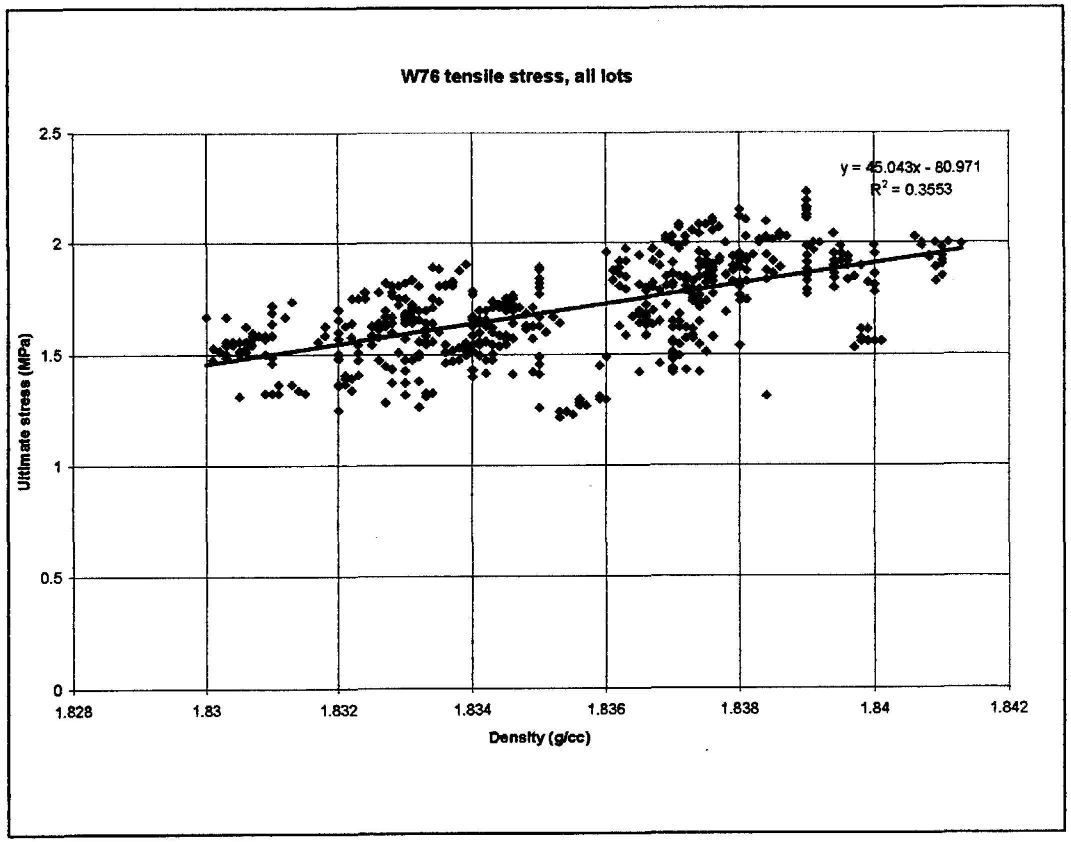
Several years ago we started a series of tests to investigate the scatter observed in surveillance tests.



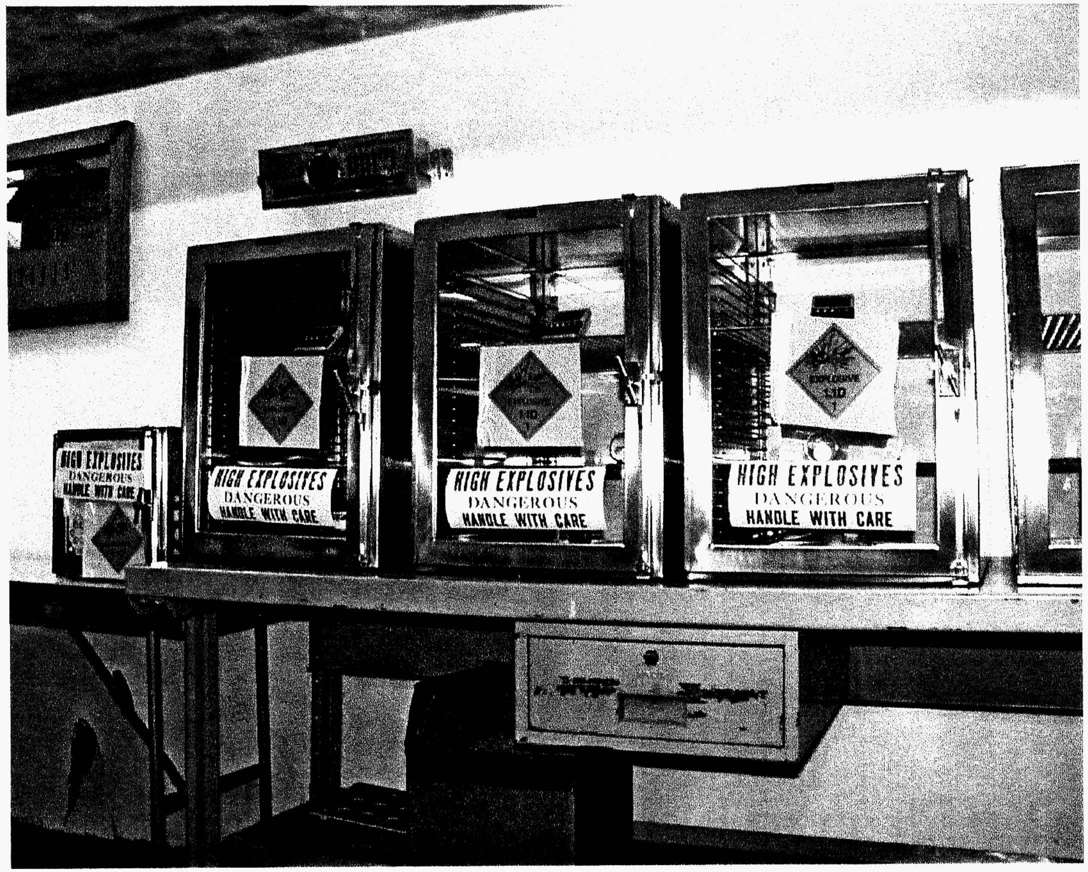


Original List of Tests

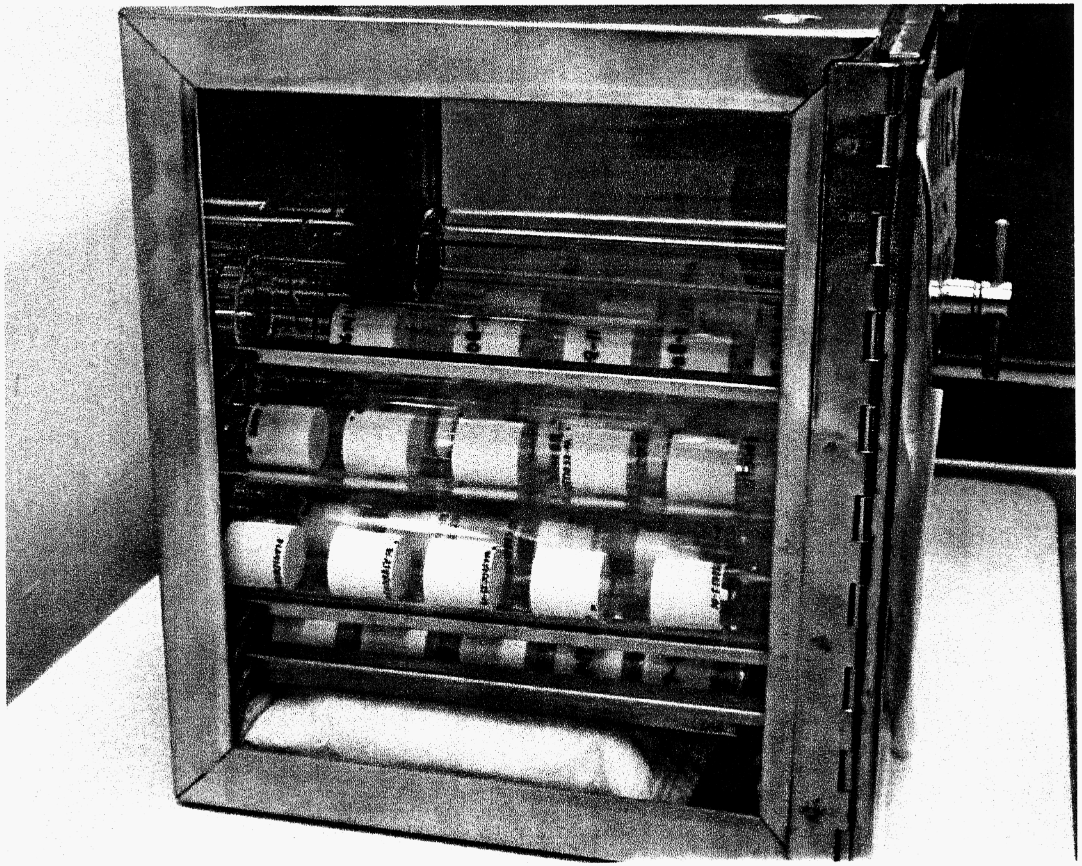
- Time since pressing
- Pressed density
- Humidity ← Main focus here
- NP migration
- Chemical aging

Humidity samples are stored in sealed cabinets

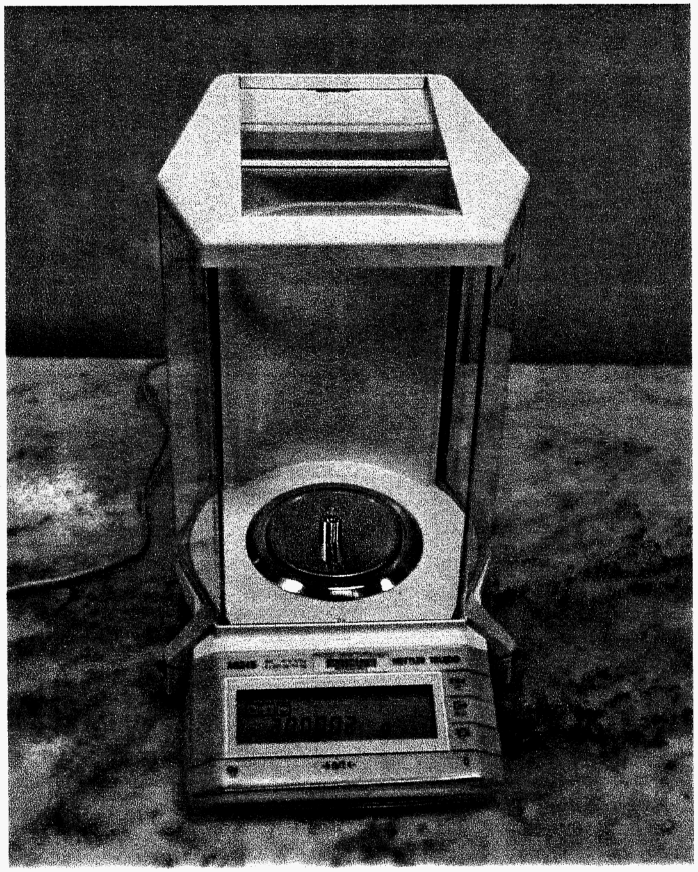
- Desiccated
- 20-25%RH (Lithium Chloride)
- 30-35%RH (Calcium Chloride Hexahydrate)
- 70%RH (Sodium Chlorate)
- 92-96%RH (distilled water)



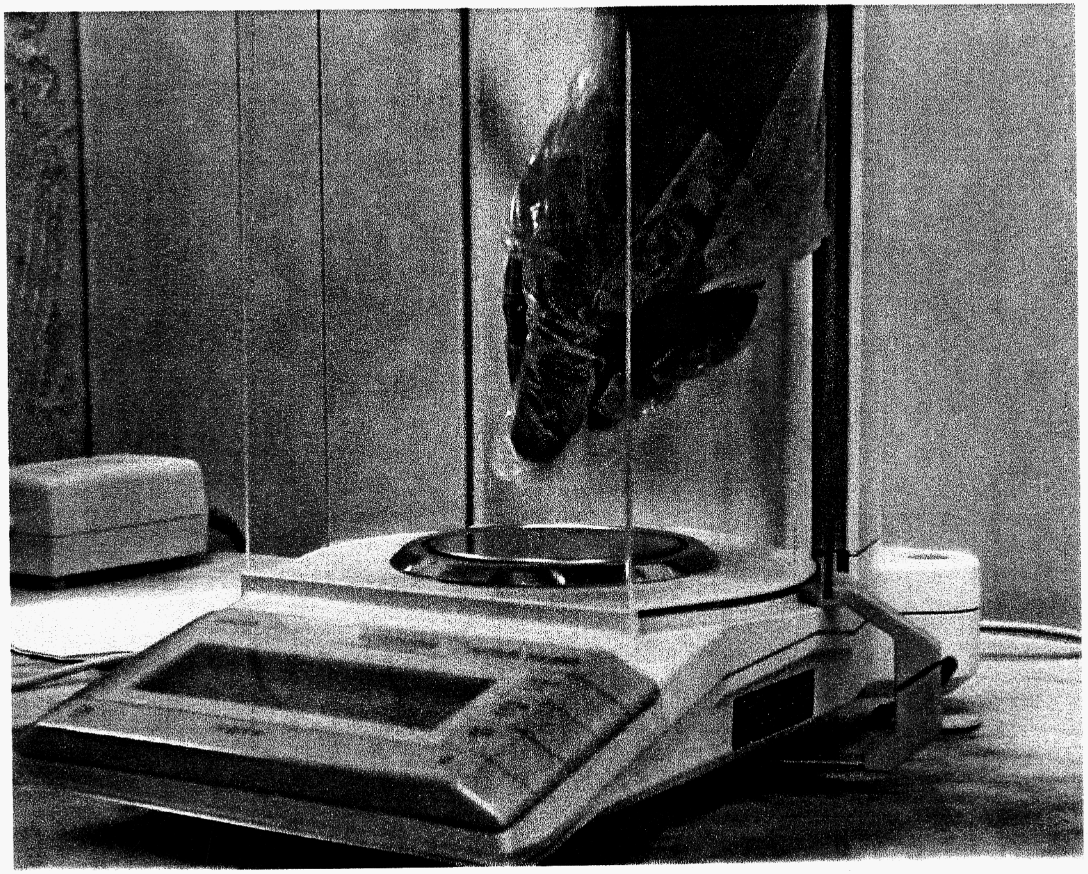
Samples are in glass tubes to avoid NP migration



Scales are verified at each weighing. Barometric pressure and room humidity recorded.

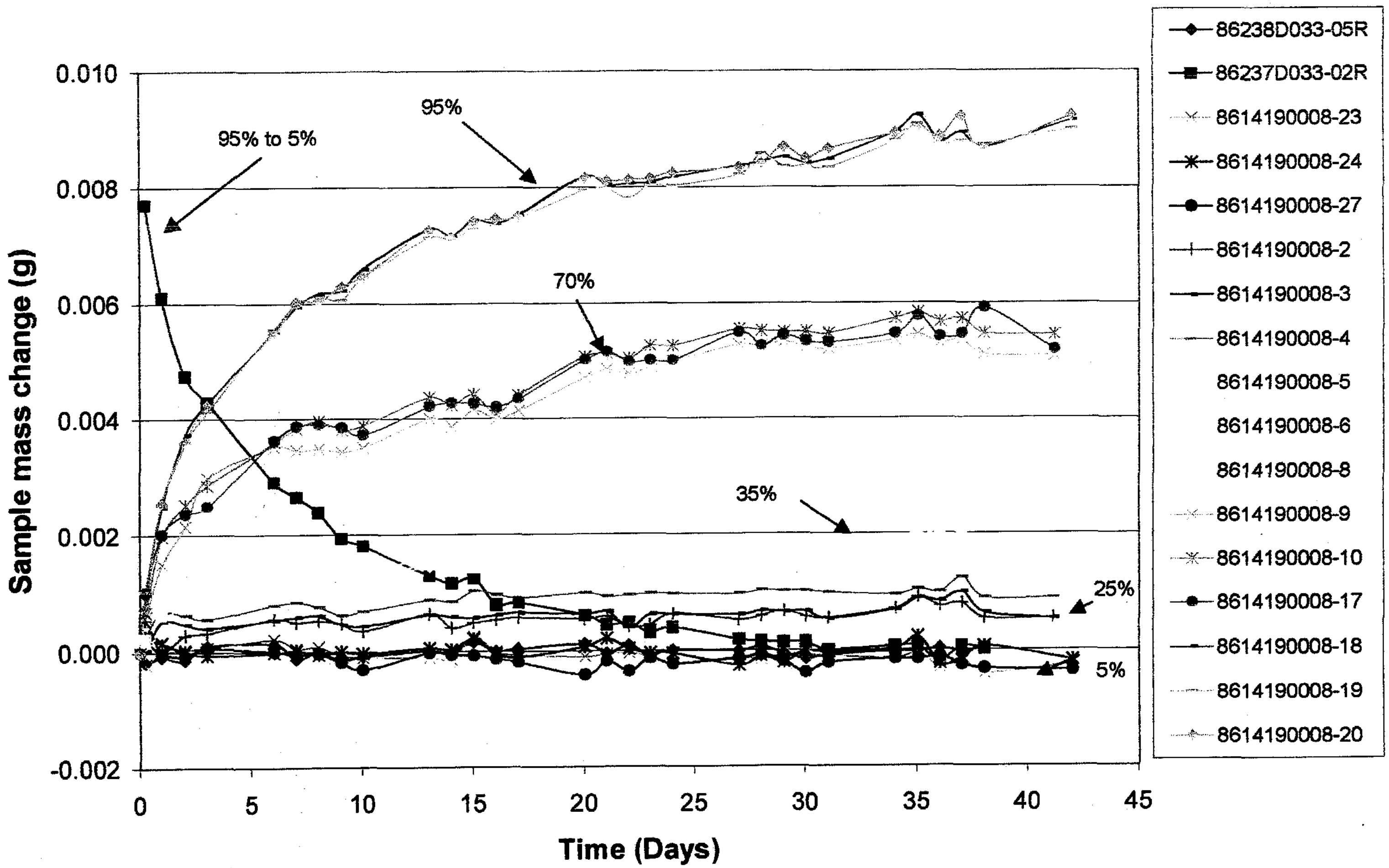


Gloves used when handling the samples to avoid sample contamination.

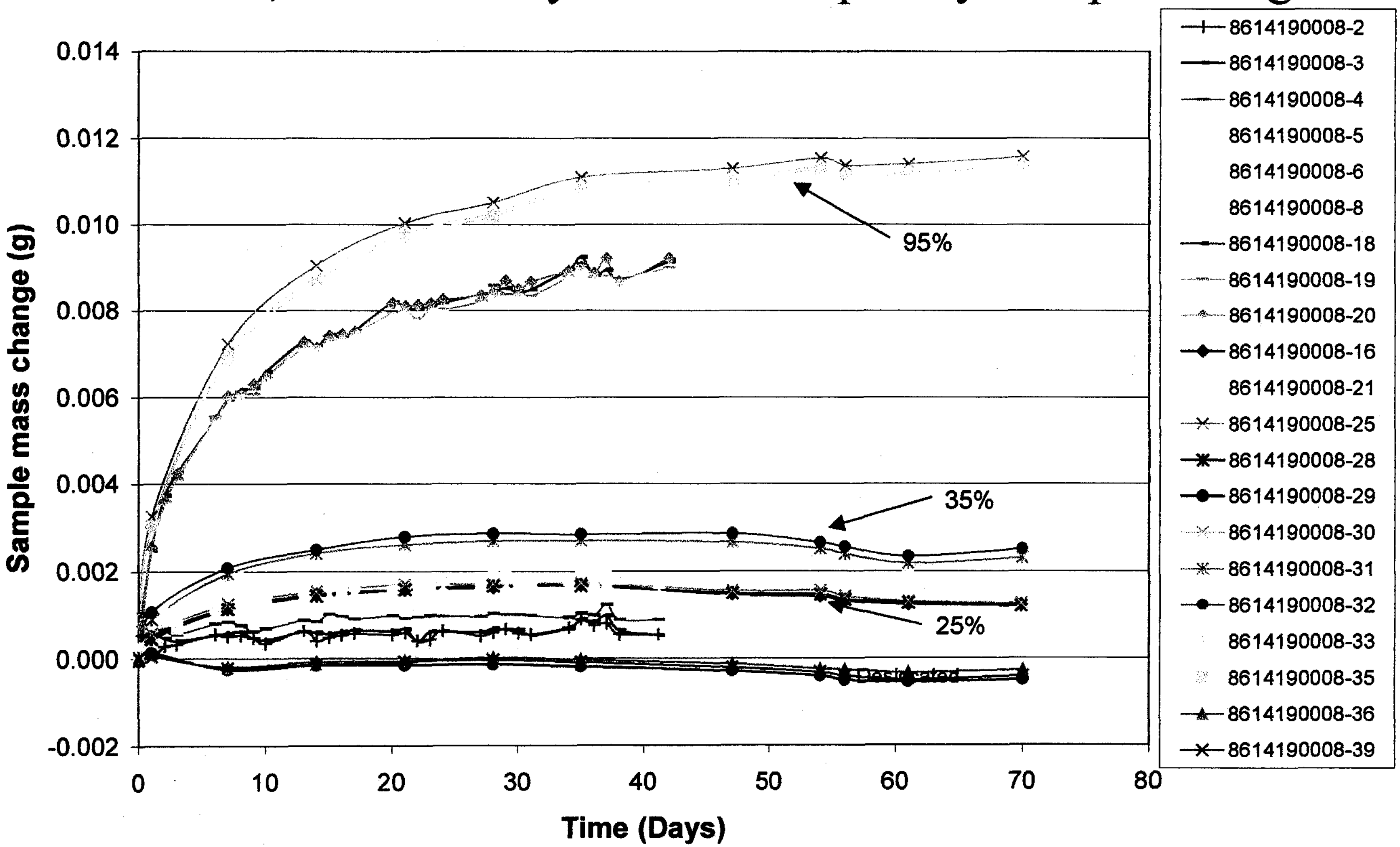


Mass recorded to 0.01 mg in latest test series, 0.1 mg in original series.

Original test series resulted in somewhat noisy but usable data. Did not get MW measurements.



Second series shows difference between daily and weekly reading schedule. Corrections for air density changes are included, but evidently are not completely compensating.



First series samples had nominal composition, except that the BDNPF content is lower than the BDNPA.

since 1
Mason & Hanger Corp

Date: July 6, 2000

From: Russell Vincent **Location:** Analytical Services 11-17

To: Tom Meyer **Location:** Applied Technology 11-2

Subject: Results for Compression Samples

Four PBX 9501 compression samples were received in 11-17 for nitroplasticizer analysis by HPLC. In addition to the nitroplasticizer analysis, the samples were analyzed for Irganox 1010 and PBNA. The samples were injected in triplicate using various mobile phases to determine the concentrations of the compounds. The averages and standard deviations reported are based on the three injections (n=3).

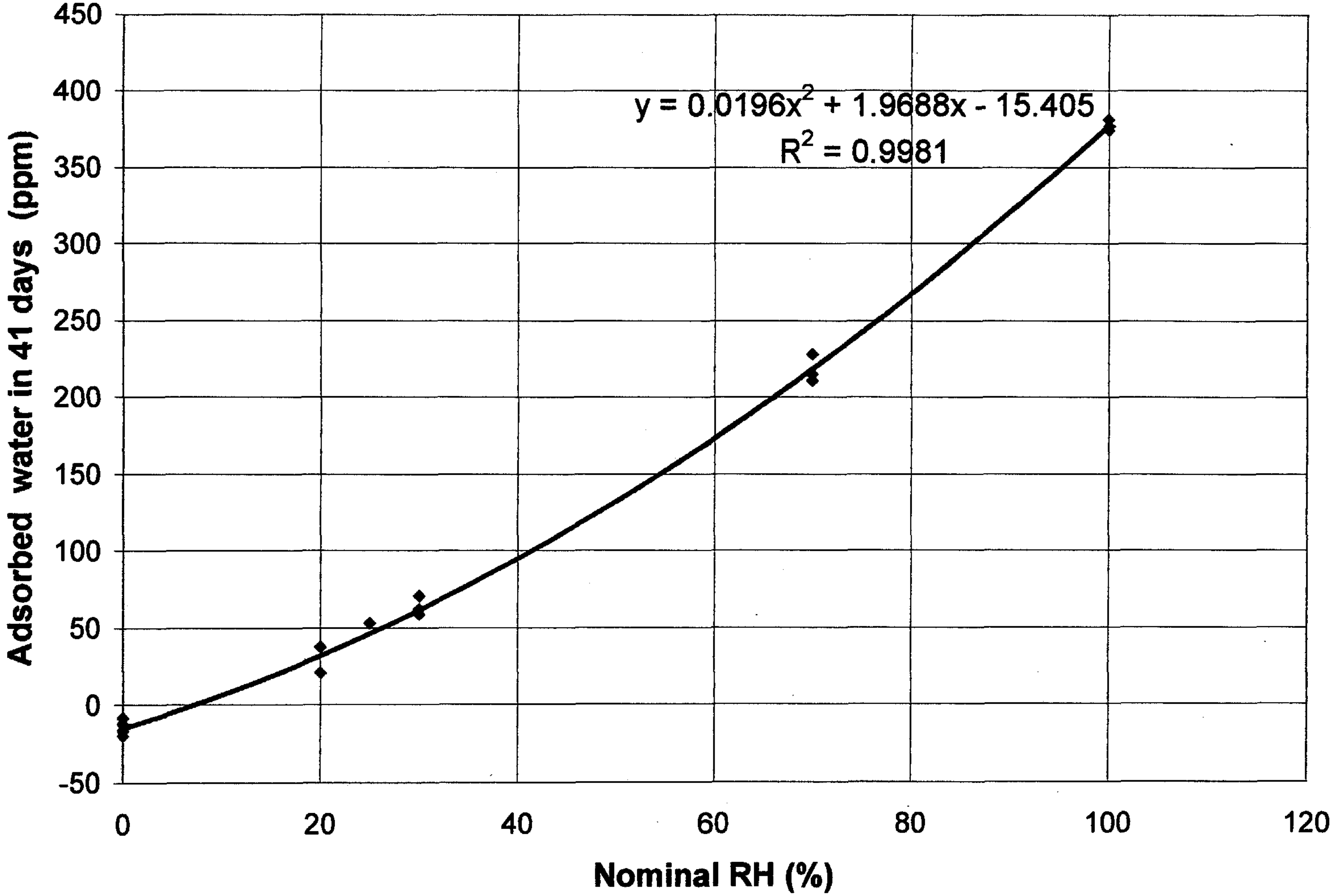
Sample ID	BDNPF (%)		BDNPA (%)	
	average	std. dev.	average	std. dev.
861419008-27	1.1111	0.0003	1.2925	0.0018
861419008-4	1.1178	0.0015	1.3002	0.0015
861419008-17	1.1246	0.0014	1.3077	0.0018
861419008-20	1.1121	0.0021	1.2938	0.0017

Sample ID	Irganox 1010 (%)		PBNA (□ g/g)	
	average	std. dev.	average	std. dev.
861419008-27	0.0617	0.0007	17.2300	0.0538
861419008-4	0.0638	0.0007	17.9214	0.0570
861419008-17	0.0612	0.0012	17.6207	0.0203
861419008-20	0.0617	0.0003	17.6884	0.0340

If you have any questions, please feel free to call me at x-4483.

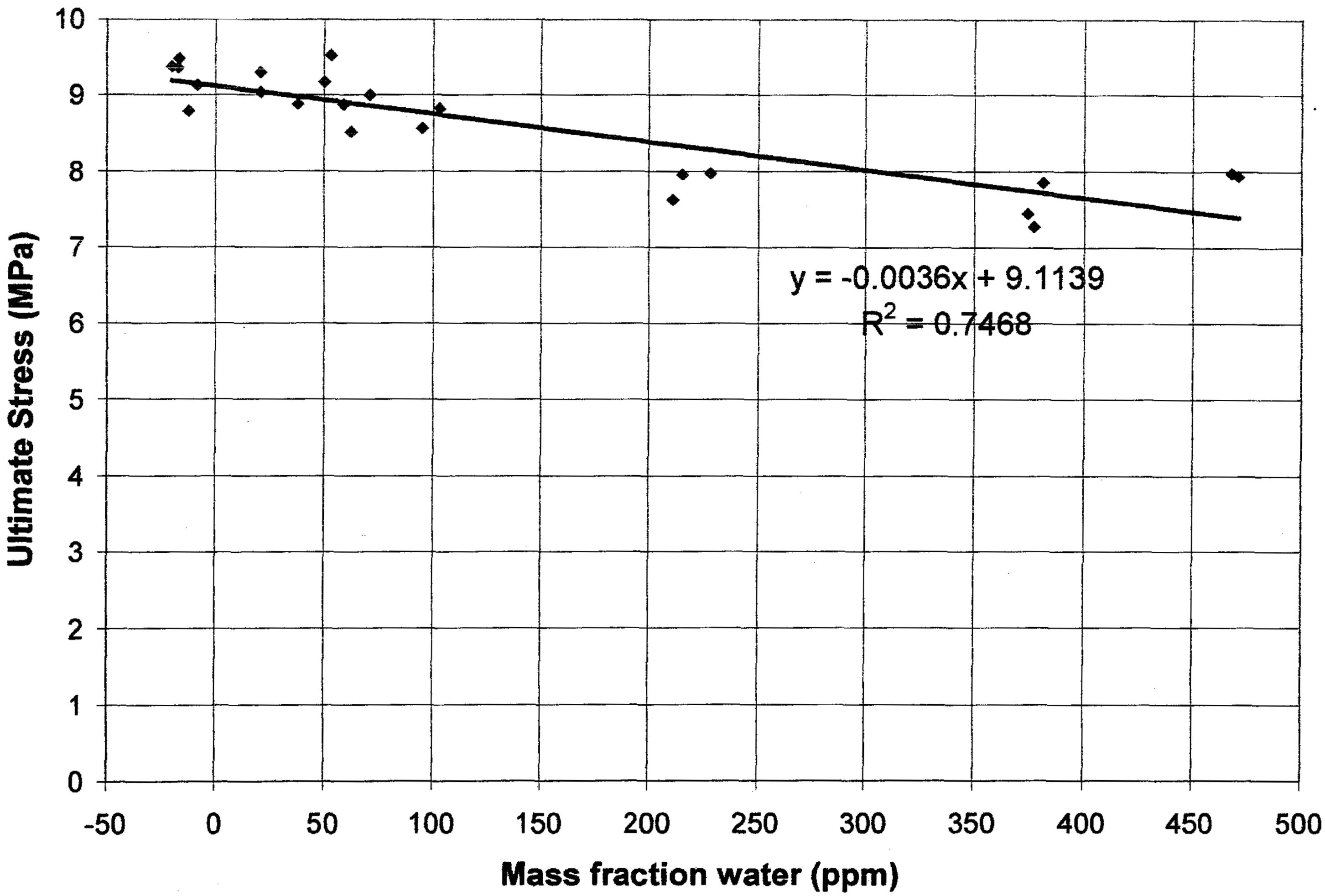
Russell Vincent
Scientist III

Change in mass shows moisture absorption.

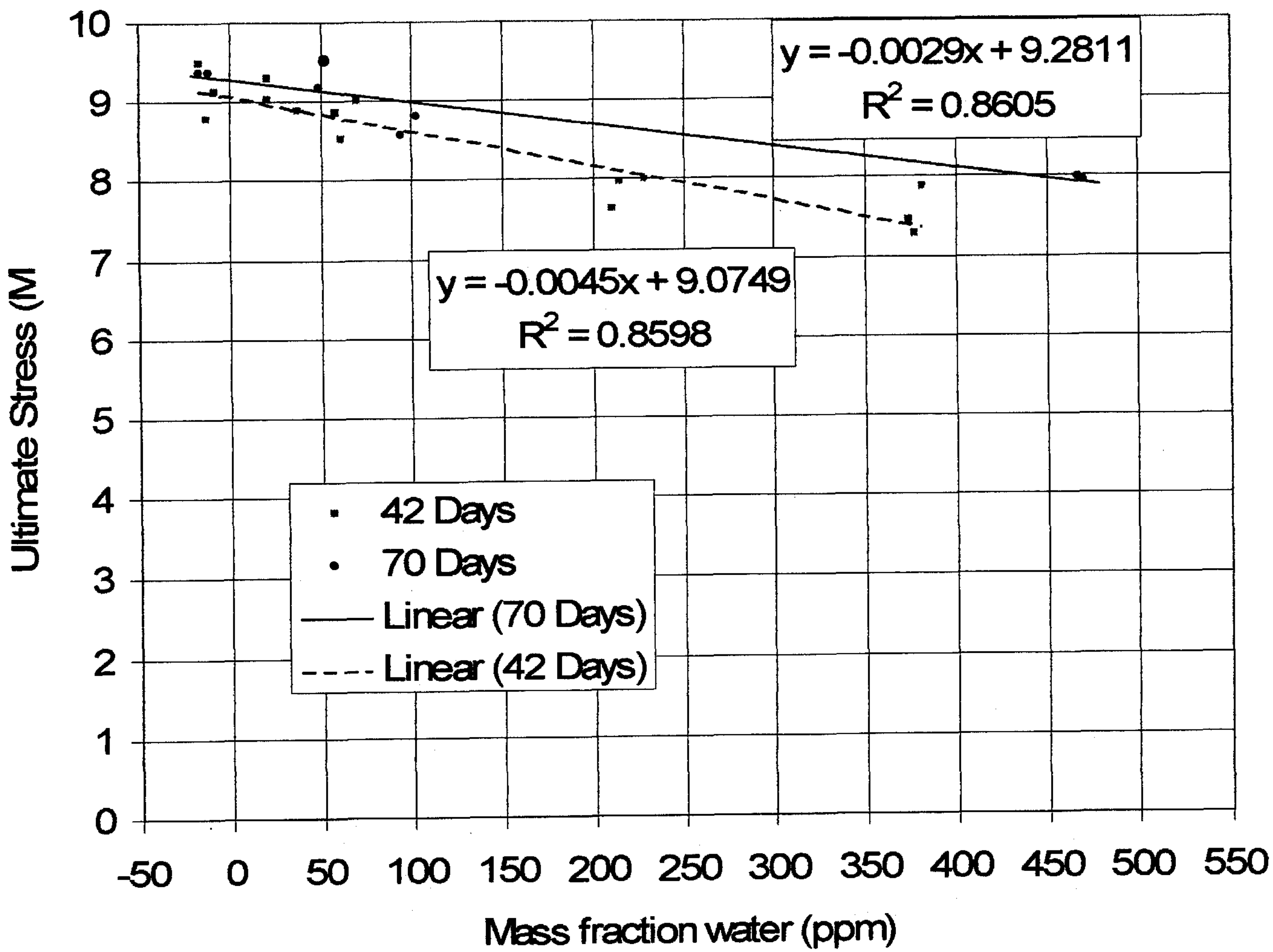


Absorbed moisture reduces compressive strength.

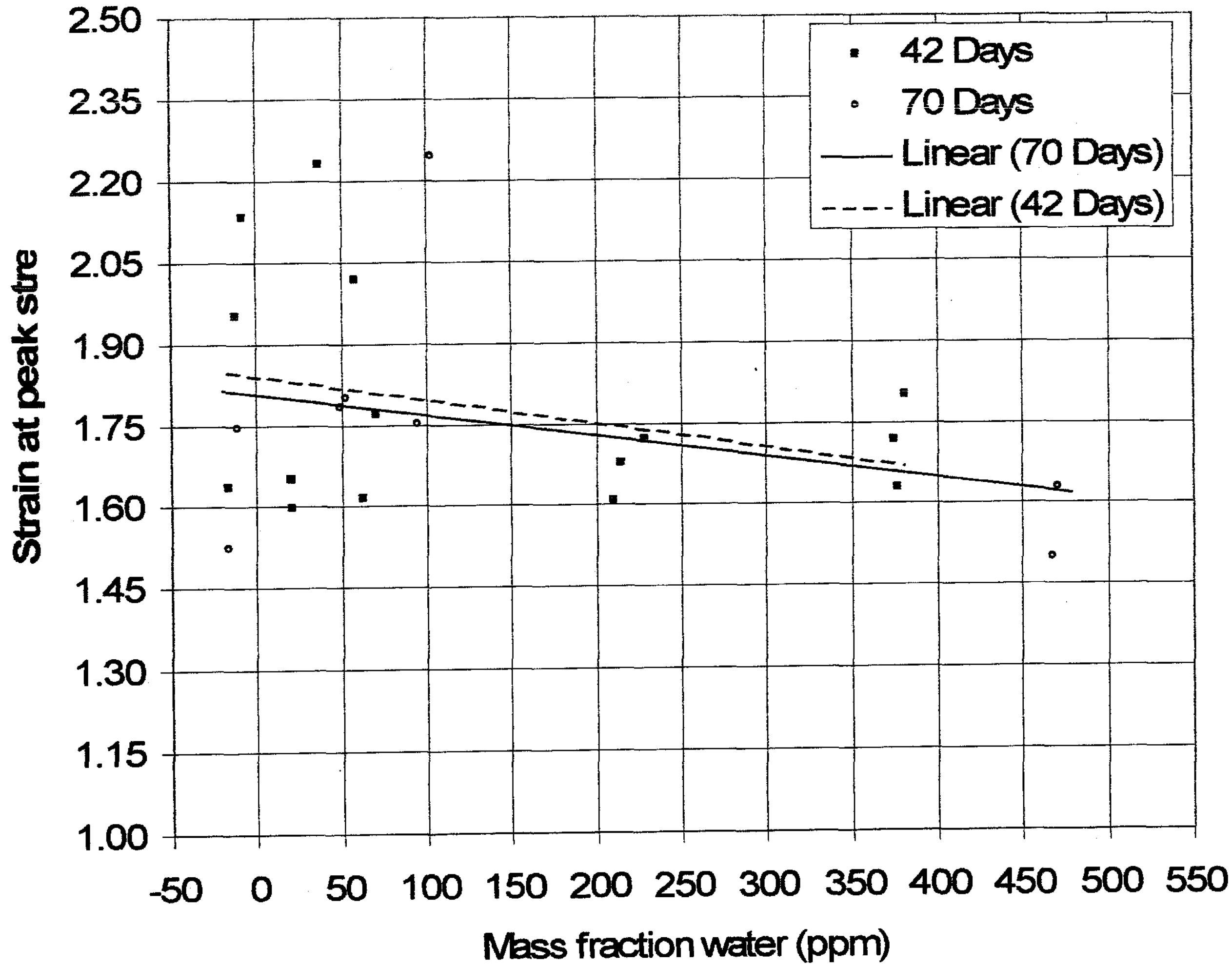
Second series values are somewhat above original series, possibly because of age or decreased NP.



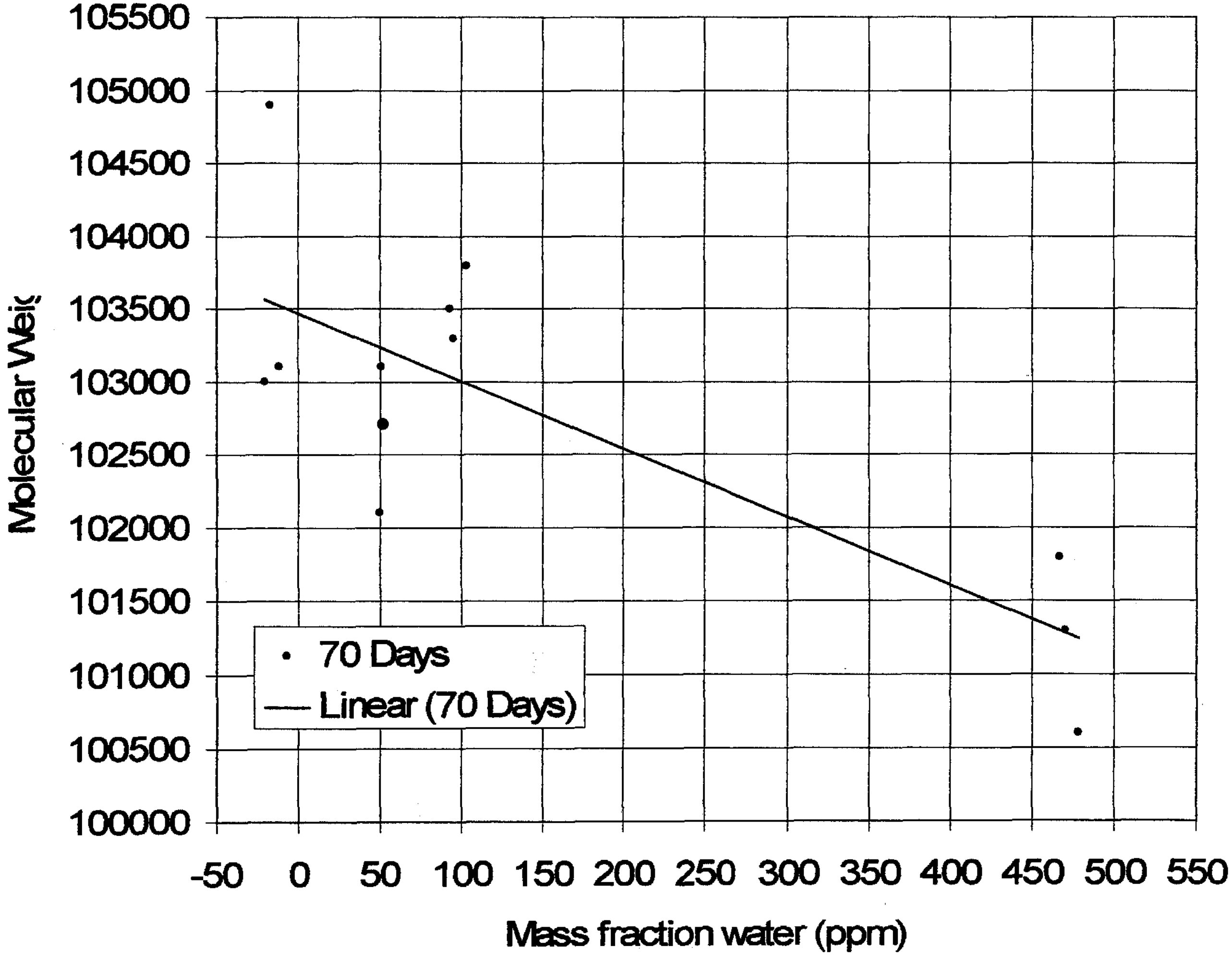
Adsorbed water reduces compressive strength II.



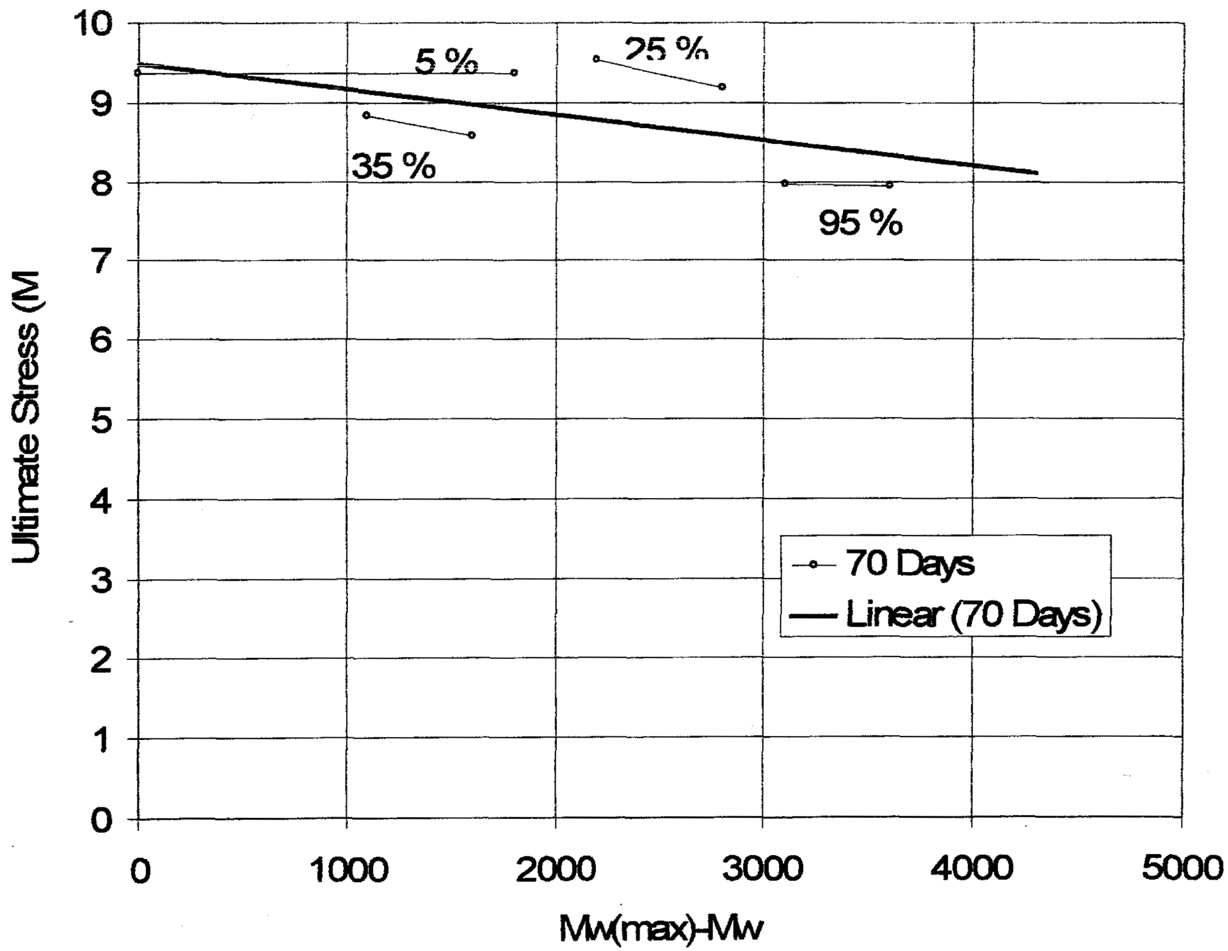
Adsorbed water reduces strain at peak stress



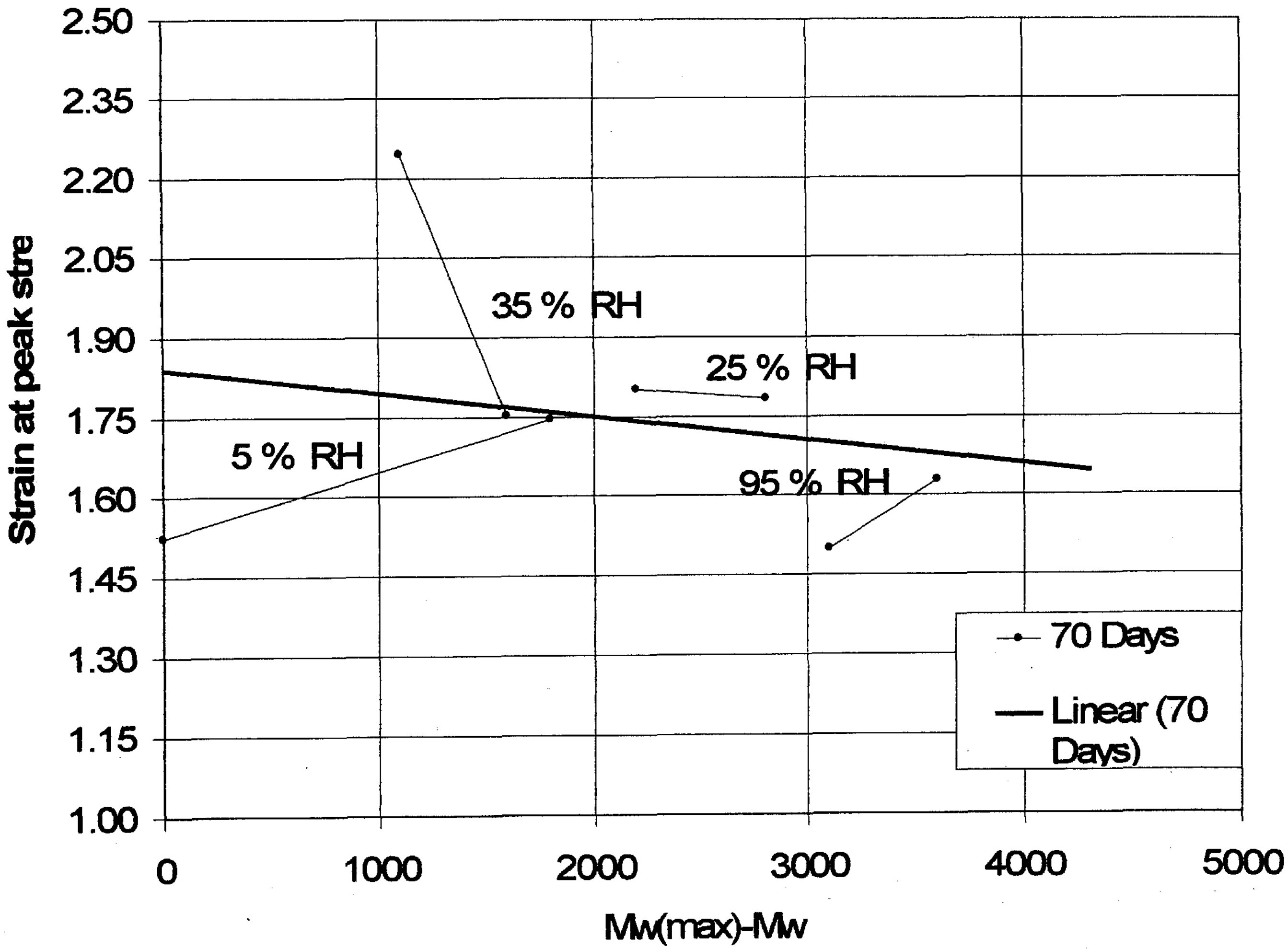
Adsorbed water reduces molecular weight



Ultimate stress decreases with molecular weight decrease



Strain at peak stress also decreases with decreasing molecular weight



Previous studies show dependence on temperature history and density effects

- Studies on neat Estane show a large change in modulus and ultimate strength properties as a function of age since thermal mixing.
- Experiments done at different times after pressing of PBX 9501 also show different behaviors. This indicates a dependence on the thermal storage conditions. The observed variations were 2-5%.
- Different lots of material press to different densities. Experiments done on a single lot of material deliberately pressed to different densities show a large variation in strength with density, but not as large an effect as observed in historic stockpile testing. It is difficult to sort out correlated effects in the stockpile data. The samples deliberately pressed to different densities and tested in compression show a 10% variation between 1.83-1.84, while the stockpile tensile data show roughly a 20% spread.
- To help reveal chemical aging effects we need a technique to compensate for the differences in density between different lots of material. Presumably test temperature, moisture content, and thermal history can be standardized.

Tentative Suggestions for Improved Surveillance Tests

- Desiccate all samples. The desiccated environment should be held through sample preparation and testing. This should be done for at least n(=2 ?) months after wet machining.
- Control temperature of all samples during storage, sample preparation and testing. Consider using a thermal standardization treatment.
- Tests done at high temperatures will likely need a very well controlled heating protocol to obtain reproducible results. The initial cooling protocol on low temperature tests should also be controlled.
- Store samples in a sealed environment made of non-absorbent materials to avoid NP loss or migration. (This might interact with desiccating procedure.)

Planned Future Tests

- Reduced NP properties (NP reduction started in Jan, first samples removed late June at about 2.3%, were just tested last week)
- Thermal history - need to redo original tests with better controls on humidity and NP content.
- Fracture behavior
  - Brazilian tests (environments,
  - Standard tensile tests (samples pressed, currently being desiccated)
- Chemical aging - Estane hydrolysis, others (?)
- Creep testing with environmental control (doing preliminary experiments at DX-2)

NP test results to date

- The first set of samples were tested August 1.
- We estimate the NP content is down by 0.2% leaving a nominal 2.3% remaining.
- Some problems with the calibration of the strain measurement occurred. We believe the loading rate was correct and the load values are correct.
- Average strength was 9.84 MPa compared to 9.17 MPa for a reference sample. This is a 7% increase in strength.

Brazil tests showing some statistical characteristics of fracture

- Working on improving imaging technology.
- New Kodak Megapixel camera on order
- Preliminary microscope images using Nikon D1

Connections with Polymer Aging LDRD

- Modeling of phase separation in Estane
  - Based on Bruce Orler's DMA data and Darla Thompson's FTIR measurements with Ed Kober's analysis.
  - Provides accurate model of time dependence of phase separation process.
- Modeling of temperature dependence of viscoelastic properties
  - More flexible than time-temperature superposition techniques
  - Can replicate very accurately entire DMA curves

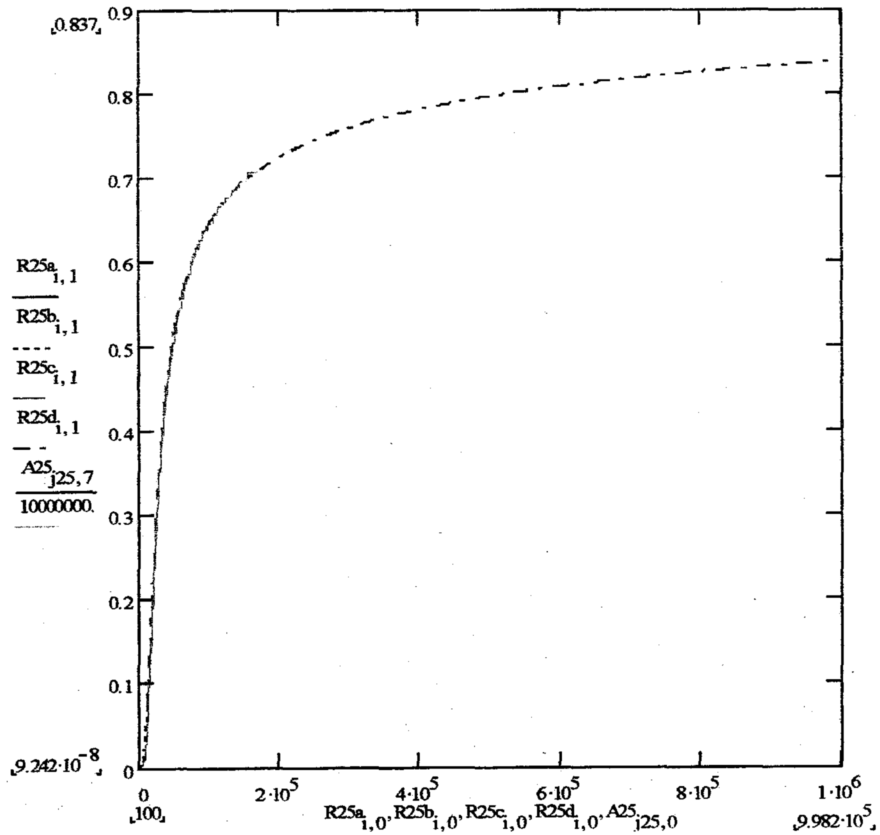
Integration of state variable model gives good agreement with original data.

This graph shows results of fitting at 25°C. Other temperatures are similar.

On linear scale the calculated fitting values (R25nn) appear to overlay the original data (A25) nicely.

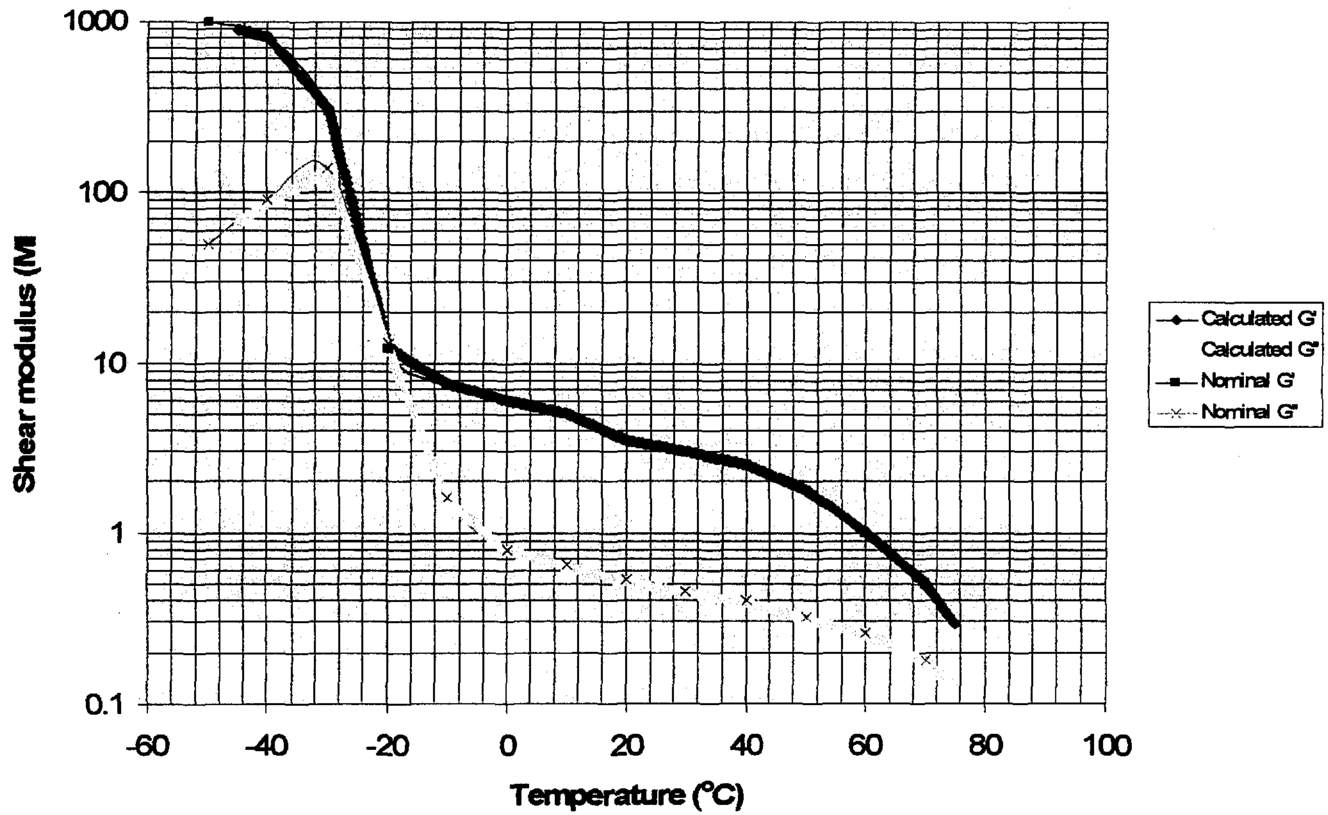
Closer inspection shows some discrepancies at small times evidently caused by small errors from the data manipulation process.

X



Time (sec)

Numerical model of dynamic mechanical analysis (DMA) experiment shows capability of Prony series technique.



Numerical model took 22000 time steps to explicitly model 0.1 Hz mechanical strain oscillations, over temperature range of -50C to +75C. The calculated model stresses, separated into phase components, and scaled to modulus values are plotted as Calculated G' and G''. Model input values of G' and G'' at discrete temperatures, plotted with a smoothed interpolated connecting line as Nominal G' and G''. The nominal values are from tests on Estane 5703.